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A PRELIMINARY EVALUATION OF DIAMOND-LIKE CARBON COATED POLYCARBONATE

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POLYMER RESEARCH BRANCH

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September 1991

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ABSTRACT

The deposition of diamond-like carbon (DLC) films on polycarbonate substrate has recently been achieved at room temperature by Diamonex, Inc. via a unique ion beam system. These diamond-like carbon coatings have considerable potential as wear-resistant protective hardcoatings for transparent armor systems. Aspects of ballistic behavior, abrasion resistance, thermal stability, and chemical resistance of the coating and any influence it has on the impact performance of the polycarbonate substrate were examined in this study. Preliminary results indicate that adhesion between the DLC coating and the polycarbonate substrate is very good. No delamination or peeling of the coating was found on the ballistically tested specimens. In addition, a well-bonded DLC coating did not cause polycarbonate to fail in a brittle fashion. Preliminary results of its inertness to the decontaminant DS2 demonstrate that the DLC coating is capable of protecting polycarbonate from abrasion while maintaining critical agent, decontaminant, and ballistic concerns.

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INTRODUCTION

There has been widespread research activity in growing diamond and diamond-like materials, and many new process breakthroughs were announced in 1990.[1-4] Currently, free-standing diamond films of thickness greater than 1 mm are produced by Norton Company.[2] The unique features of synthetic diamond materials are outstanding mechanical hardness, wear resistance, chemical inertness, thermal conductivity, and thermal stability. In addition, diamond products are transparent to visible light, X-ray, ultraviolet radiation, and much of the infrared spectrum.[1] Such properties have potential in fabricating wear-resistant protective coatings, anti-reflective coatings, abrasives, high temperature semiconducting and optical devices, and heat sinks.[1,2,5] The market for diamond films is reported to be about $200 million in 1991 and is expected to exceed $2 billion by 1995 and $4 billion by 2000, according to a market study by International Resource Development.[6]

The focus of many recent research efforts is aimed at producing diamond-like carbon (DLC) films on temperature-sensitive substrates.[3-5] The latest development of significance is the demonstration by Diamonex, Inc. that DLC coatings have been successfully deposited on polycarbonates, via a unique dual ion beam system. In this process, which was originally developed by NASA, an ion beam of controlled composition, energy, and flux is directed onto a substrate. Another low energy ion beam is used to clean the surface without damaging the underlying substrate, as well as to remove weakly bound physisorbed gases or impurities.[3] DLC coatings presumably consist of carbon atoms in chemical structures similar to that of diamond but without long-range crystal order.[3]

DLC coatings will play an important role in transparent armor applications such as lenses for individual protection and vision blocks used in combat vehicles. Currently, polycarbonate is being widely used based upon its optical clarity, toughness, and resistance to fragment penetration. Unfortunately, polycarbonate has deficiencies in its abrasion and solvent resistance. Numerous studies have been carried out on alternate transparent polymers,[7-9] as well as on various protective hardcoatings.[10] However, there has been no significant progress in achieving optimum optical, thermal, mechanical, and chemical properties.

The objective of this study was to evaluate diamond-like carbon coated polycarbonates. Aspects of ballistic behavior, abrasion resistance, thermal stability, and chemical resistance of the coating and any influence it has on the impact performance of the polycarbonate substrate were examined. This document reports the preliminary test results, as well as the micrographs of scanning electron microscopy (SEM) studies.

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EXPERIMENTAL

Materials

The injection molded polycarbonate coupons of 2.3 mm (0.09 in.) thick, coated with DLC on one or both sides, were purchased from Diamonex, Inc. The DLC coating is ion beam deposited with a thickness of about 0.1 μm. All the DLC coated polycarbonate coupons possess another base coating, a proprietary polysiloxane compound developed by Gentex, Inc. and Diamonex, Inc. The polysiloxane coating was utilized to enhance adhesion between the DLC coating and the polycarbonate. Polycarbonate samples with only Gentex coating, the base coating, were also evaluated.

Test Methods

Ballistic Impact

Ballistic testing was carried out using the standard .22 caliber fragment simulator projectile weighing 1.10 grams. The projectiles were fired from a four foot long air gun which is connected to a high speed solenoid valve leading to a helium gas cylinder.[8] Before firing, a desired pressure was selected, then the gun was fired by manual closure of an electrical circuit which opened the solenoid valve. The projectile velocities were determined by a pair of printed silver grid-paper screens located in front of the specimen and connected to an electronic chronograph for time-of-flight measurements. Specimen impact responses were rated in terms of a characteristic projectile impact velocity V_{50}. This quantity is defined as the projectile impact velocity at which there is 50% probability of complete penetration of the target specimen. The higher the value, the more resistant the specimen is to projectile penetration.

Taber Abrasion

The Taber abrader was utilized following the ASTM D 1044-85 method.[11] The apparatus consists of a rotating platen, upon which the sample is mounted, and a pair of abrasive wheels resting perpendicular to the sample surface. The sample is rotated, causing the abrasive wheels to rotate in opposite directions, thus abrading the sample surface. A vacuum nozzle is positioned just above the sample surface to remove debris caused by abrasion. The weight chosen in this study was 500 g. After samples were abraded to a selected number of revolutions, the intensity of damage was quantified by measuring the percentage of haze with an integrating sphere haze meter according to the ASTM D 1003-61 method.[12]

Sand Blasting

A sand blasting test method was utilized[13] where 40 ± 1 grams of silica grit were projected at a substrate area of 8.06 cm^2 (1.25 $in.^2$) over a distance of 8.9 cm (3.5 in.). The damage was assessed similar to the above by measuring the increase in haze.

11. ASTM. *Standard Method of Test for Resistance of Transparent Plastics to Surface Abrasion*. ASTM Designation D 1044-85, 1988, p. 380-383.
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Chemical Exposure

Samples were exposed to drops of various liquids at room temperature, including chloroethylsulfide (CEES), a simulant for the blister agent HD, and the universal decontaminant DS2. The drops were applied either directly to the substrate surface or to a small piece of filter paper on top of the substrate to prevent spreading of the liquids. In some cases, the samples were covered by an inverted watch glass to retard evaporation. Surfaces were examined for deterioration visible to the unaided eye.

Scanning Electron Microscopy

Damaged areas of test specimens were examined using an AMRAY scanning electron microscope. A thin gold palladium coating was evaporated on the surface to reduce charging effects under the electron beam.

RESULTS AND DISCUSSION

Optical Clarity

A typical DLC-coated polycarbonate coupon with a coating thickness of about 0.1 μm is shown in Figure 1. It is transparent but with a brownish tint. The intensity of the color increases as the coating thickness increases. Optical transmission was measured to be 80% for DLC-coated polycarbonate compared to 87% for uncoated polycarbonate.

Figure 1. A typical DLC-coated polycarbonate coupon is transparent, but with a brownish tint.

Ballistic Impact

The V_{50} value obtained for the DLC-coated polycarbonate specimens of 2.3 mm (0.09 in.) thick is 195 m/sec (640 ft/sec), close to the V_{50} obtained for the uncoated PC specimens at 196 m/sec (643 ft/sec). These V_{50} values are similar to those obtained for the untreated

olycarbonate by Lewis and Parsons.[14] The V_{50} values for the DLC-coated PC are consistent, regardless of being coated on one side or on both sides. Results also show similar V_{50} values when single-side coated specimens were impacted from either the coated or uncoated side.

The type of response to the projectile impact was also examined. Figure 2 shows a ypical double-side DLC-coated PC specimen after multiple ballistic impact, which exhibits various damage zones including complete, partial, and no penetration. No flaking or peeling of the coating was noticed. In addition, a significant difference in appearance between he DLC-coated and uncoated polycarbonate specimens was observed as a result of the mpact. On each DLC-coated specimen, extensive damage of the coating was observed around the areas of impact. This is true for either single-side or double-side coated specimens; as a result, the effected regions of the DLC-coating caused the scattering of the light. In addition, fringe patterns were observed, under the polarized light, within the damage area, as shown in Figure 3.

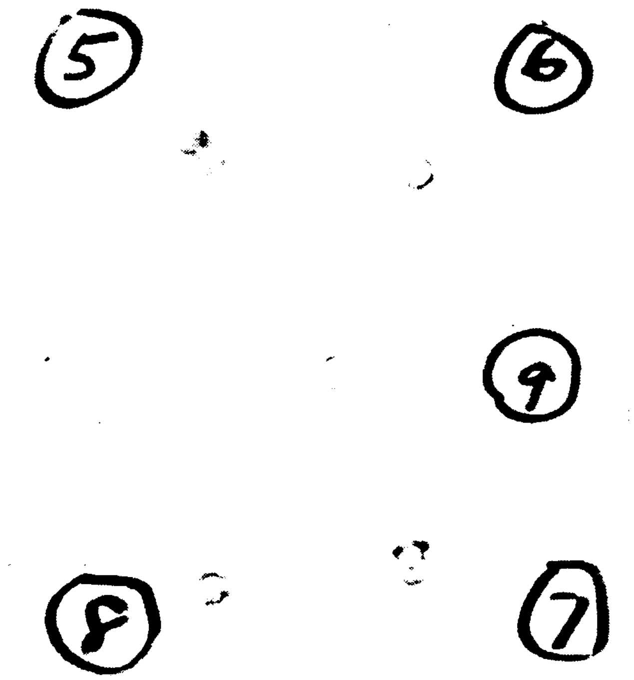

Figure 2. A DLC-coated polycarbonate specimen exhibits various damage areas after multiple impacts; complete penetration in #6 and #9, partial penetration in #7, and no penetration in #5 and #8.

14. LEWIS, R. W., and PARSONS, G. R. *Ballistic Performance of Transparent Materials for Eye Protection (U).* U.S. Army Materials Technology Laboratory, AMMRC TR 72-36, November 1972 (Confidential Report).

Figure 3. A ballistically-impacted polycarbonate specimen, same as in Figure 2, except taken under the polarized light.

On a closer examination via SEM, a fine network of microcracks were present around the areas of impact, as shown in Figures 4a and 4b. These cracks appeared to propagate only on the coating layer without penetration into the polycarbonate substrate.

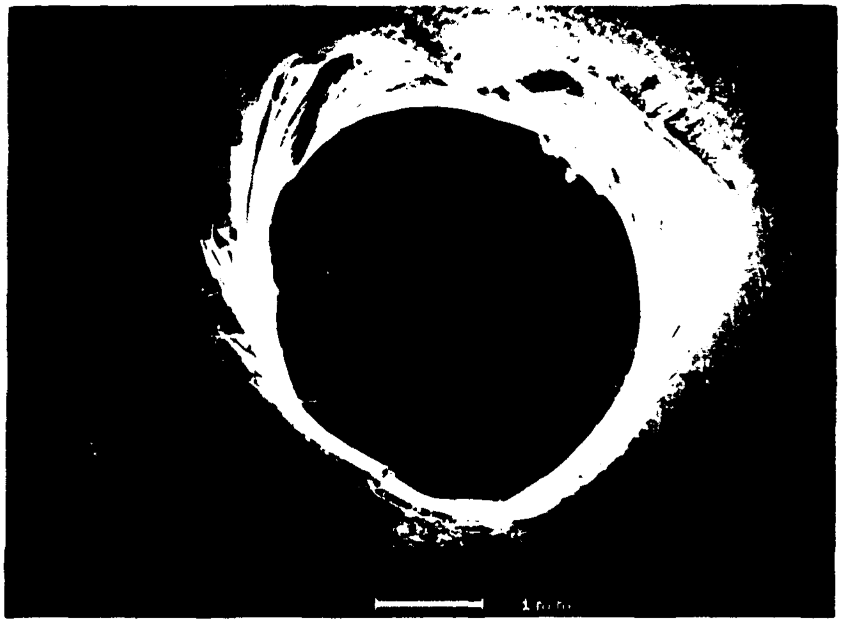

Figure 4a. SEM micrograph reveals a region of extensive damage around the area of impact.

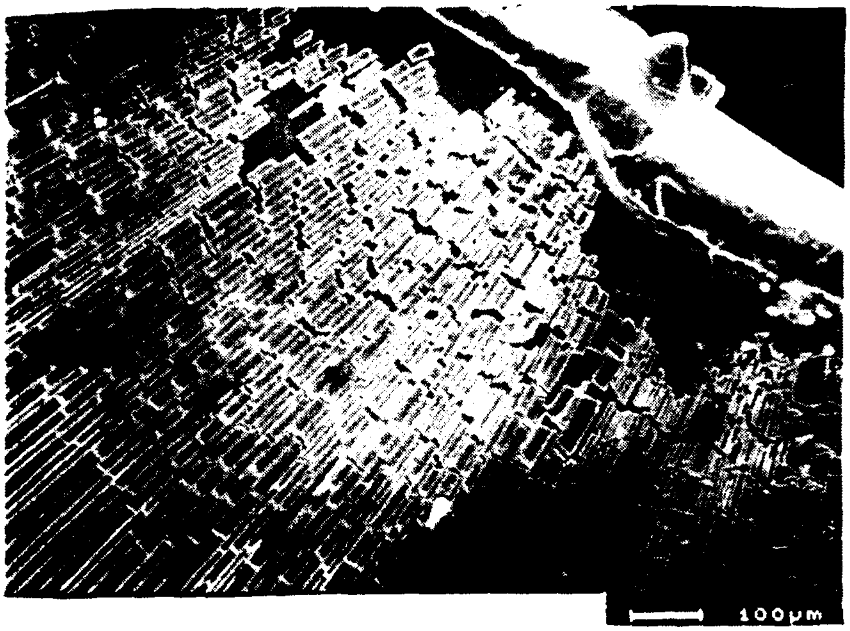

Figure 4b. SEM micrograph shows a fine network of microcracks within the damage area at a higher magnification.

In short, DLC coating applied via an ion beam process provides good adhesion to polycarbonate substrate while maintaining the ballistic performance. Extensive microcracking on the coating occurred as a result of the ballistic impact.

Abrasion Tests

Resistance to abrasion is one of the important criteria in materials selection for transparent armors. Taber abrasion has often been used to evaluate the hardness of the coating. However, the effect of sand abrasion on armor performance is of considerable interest to the military due to recent events in the Persian Gulf.

The nature of sand blasting is different from abrasion by a Taber abrader; sand blasting is characteristic of dynamic impact and Taber abrasion is a static scratch test. In practice, Taber abrasion favors coatings with higher hardness, while dynamic impact abrasion yields better results with less brittle (or more compliant) coatings. However, measurements of the resistance to abrasion in both tests strongly depend upon the rigidity of the substrate.

Taber Abrasion

Preliminary results obtained for specimens abraded after 100 revolutions are summarized in Table 1. Values of the increase in haze readings for the DLC-coated polycarbonate are comparable to that for the Gentex coating, and both coated specimens show better resistance to Taber abrasion than the uncoated polycarbonate. Tests for extended abrasion and different weights are in progress.

Table 1. TABER ABRASION DATA ON POLYCARBONATES

Material	Original Haze (%)	Increase in Haze (%)
Uncoated Polycarbonate	0.4	19.9
Polycarbonate with Gentex Coating	0.9	0.3
Polycarbonate with DLC Coating	0.2	0.2

Sand Blasting

The objective of sand blasting is to provide a comparison of material performance between coated and uncoated polycarbonate. The haze readings obtained from the abraded samples tested at two different sand impact velocities are listed in Table 2. Polycarbonates with either DLC coating or Gentex coating have similar properties, which are better than those of the uncoated samples. Measurements of the exact impact velocities were not available at the time of testing; however, the range of these velocities were wide enough to distinguish the material response. Figure 5 gives a comparison of DLC-coated polycarbonates before and after sand blasting.

Table 2. SAND BLASTING DATA ON POLYCARBONATES

Material	Original Haze (%)	Increase in Haze (%)	
		At Low Impact Velocity	At High Impact Velocity
Uncoated Polycarbonate	0.4	53.5	77.5
Polycarbonate with Gentex Coating	0.9	22.8	53.2
Polycarbonate with DLC Coating	0.2	19.2	50.6

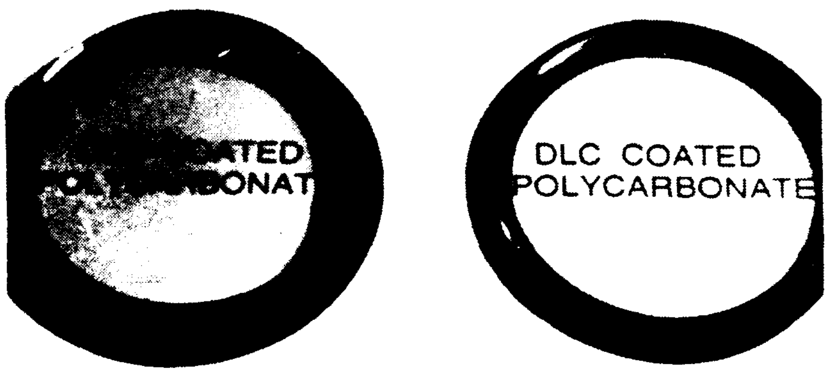

Figure 5. DLC-coated polycarbonate specimens before (on the right) and after (on the left) sand blasting.

In summary, DLC coating exhibits adequate performance in both abrasion tests suggesting that in addition to the hardness its toughness is a contributing factor.

Chemical Exposure

DLC- and Gentex-coated polycarbonate and uncoated polycarbonate samples were each exposed to droplets of DS2 and CEES at room temperature. Shortly after application to the DLC-coated sample, the drop of liquid, either DS2 or CEES, had spread to a large area. The droplets on the other two samples had spread but to a lesser extent.

No visible effects were observed when the DLC- and Gentex-coated polycarbonate samples were exposed to CEES. In the case of DS2, there was a significant reduction in clarity of the uncoated polycarbonate sample which can be attributed to dissolution and craze formation. Microcracks were observed on the Gentex coating indicating its susceptibility to attack by DS2. No visible effects were observed on the DLC-coated sample with DS2.

Thermal Stability

Thermal stability of the DLC-coated PC was examined by heating a coated specimen to 120°C (248°F) in an oven for a total of four and one-half hours. No visible damage to the DLC coating was observed.

CONCLUSIONS

DLC coating provides polycarbonate with improved abrasion resistance. The DLC-coated PC exhibits comparable ballistic strength as compared to standard PC. A well-bonded DLC coating did not cause polycarbonate to fail in a brittle fashion. No delamination or peeling of the coating was found on the ballistically tested specimens. Instead, microcracks of the DLC coating resulted. In addition, its inertness to DS2 demonstrates that this DLC coating is capable of providing desired optical, ballistic, and chemical properties.

Another application for DLC coating that is being pursued is to increase the wear, abrasion, and chemical resistance of the current ballistic/laser protective spectacles (BLPS) system. In addition, deposition of the DLC coating as a moisture barrier over either dielectric or holographic coating will be explored; these moisture sensitive coatings provide good protection against laser induced damage. Proper adhesion between each coating layers needs to be established for such multilayer coating systems.

The stability of DLC-coated polycarbonate at 120°C indicates the potential application for the laminated transparent armors, in which the structure parts including a DLC-coated PC are required to be bonded between 80°C and 120°C in an autoclave.

The current DLC coating capacity at Diamonex, Inc. can hold six pieces of eight-inch diameter coupons, and two additional deposition apparatus will be available in the near future which can hold parts in size up to either 24-inch square or 16-inch diameter, respectively.

Further studies of the DLC coatings will be carried out including characterization on the DLC films via Raman spectroscopy, determination of environmental durability of the films via exposure to UV radiation, heat, moisture, and analysis of the fracture morphology.

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